WO 2005/004646 PCT/IB2004/002222

# PROCESS FOR OBTAINING TOBACCO LEAVES WITH A STANDARDIZED NICOTINE CONTENT AND/OR IMPROVED COMBUSTIBILITY

#### FIELD OF INVENTION

This invention relates to a process for the treatment of tobacco leaves, in particular a process for obtaining tobacco leaves with a desired, standardized nicotine content and improved combustibility.

### 5 PRIOR ART

10

20

Obtaining tobacco with a constant nicotine content is important in order to guarantee the manufacture of standardized cigarettes with a fixed, constant nicotine content batch by batch, regardless of the biological variability of that content. The leaves of the Virginia variety of tobacco have a nicotine content ranging from a minimum of 0.7 to a maximum of 2.5%.

Cigarettes with a pre-determined nicotine content are currently manufactured by various methods, which are usually complex and expensive, and involve the various techniques listed below, possibly in combination, which always start with the cured tobacco leaves:

- the use of leaves with a low nicotine content, obtained by varietal selection and/or on the basis of their position on the plant (the median and lower median leaves are generally used);
  - the use of the vertical ribs, which have a very low nicotine content and are mixed with the leaves to dilute their nicotine content. In order to use them without pulverization, however, they must be processed by rolling (passage through rollers which presses them thinly so that they can be shredded) or by expansion (an expensive technique that involves subjecting the ribs to high vacuum until they "burst", thus providing shreddable material);
  - the use of regenerated tobacco, ie. tobacco powder deriving from

WO 2005/004646 PCT/IB2004/002222

manufacturing waste, mixed to a paste with cellulose and subsequently rolled and shredded;

2

blending of various types of tobacco with different nicotine contents.

5

10

20

25

A Japanese patent application dated 29 May 1972 (JP 51-16518) claims a method for the treatment of tobacco leaves which comprises the extraction of raw tobacco leaves with an organic solvent or a mixture of organic solvent and water, recovery and drying of the extracted leaves, and finally heat treatment, so that the leaves take on the required colour. Said treatment eliminates the traditional curing stage, thus accelerating the overall process, but without achieving the aim of standardizing the nicotine content of the tobacco leaves, still less their combustibility. Moreover, the extraction is performed on the raw leaves, not previously subjected to drying and curing, these operations being eliminated by the present method.

Apart from the nicotine content, the chlorine content is particularly important, as it significantly influences the combustibility, flavour and texture of the leaves.

Combustibility, ie. the ability of tobacco to remain lit once combustion has begun, is the fundamental requirement of smoking tobacco, on which all its other characteristics depend.

Tobacco leaves with a low chlorine content (the leaves of the Bright variety of tobacco have chlorine values which must not exceed 1% of the dry matter) are therefore preferred.

The use of fertilizer with a low chloride content produces tobacco with a low chloride content; however, the chloride content in the leaves is influenced by other factors which it is difficult to modify, such as the characteristics of the soil, irrigation water, etc.; moreover, the chloride level varies according to the position of the leaves on the plant (difference between basal, median and apical leaves), which means that some types of leaves have to be rejected.

5

10

15

20

25

The present process reduces the chloride content, and therefore transforms tobacco leaves with very low or nil combustibility into leaves with ideal combustibility.

# **DESCRIPTION OF THE INVENTION**

It has now been found that if the extraction is performed on previously cured, dried leaves, tobacco leaves with the desired nicotine content can be obtained, depending on the extraction conditions; the process also reduces the chloride content and consequently transforms tobaccos with little or no combustibility into tobaccos suitable for the production of cigarettes and other smoking products.

This process consequently produces tobacco leaves with a predetermined, standardized nicotine content from a raw material (tobacco leaves) with a variable nicotine content (0.7-2.5%). The process also reduces the chloride content, which adversely affects the combustibility, texture and flavour of the leaves.

This invention relates to a process for the treatment of tobacco leaves (Nicotiana tabacum) comprising the following steps:

- a) drying and curing of the leaves;
- b) extraction of the dried leaves with a solvent or mixture of solvents;
- c) re-drying of the extracted leaves;
- d) elimination of the ribs.

The extraction solvent can be selected from methanol, ethanol, propanol, isopropanol, acetone, ethylene glycol and water; they can be used individually and/or in appropriate mixtures, and also with the pH suitably adjusted with buffer solutions. A water/alcohol mixture with a strength of between 20° and 80° is preferably used, and even more preferably an ethanol/water mixture with a strength of 50°.

The weight ratio between the tobacco leaves and the solvent mixture is

5

10

15

20

**25** .

between 1:5 and 1:30, and preferably between 1:10 and 1:20; even more preferably, the ratio is 1:16 to reduce the nicotine content by 80%, starting from tobacco leaves with a 1% nicotine content.

The extraction time ranges between 3 and 16 hours, preferably 8 hours, whereas the extraction temperature is between 20 and 80°C, preferably 60°C, to reduce the nicotine content by 80% starting from tobacco leaves with a 1% nicotine content.

The number of extractions performed ranges between 1 and 5, in order to obtain, preferably, an 80% reduction in the nicotine content, starting from tobacco leaves with a 1% nicotine content.

The drying time of the extracted tobacco leaves is between 36 and 48 hours, and the drying temperature is 35°C.

The tobacco leaves thus obtained, which also form the subject of this invention, can then be stored pending the subsequent stages of processing to make cigarettes, cigars or shags. In their manufacture, tobacco leaves must be beaten to eliminate the ribs and shredded to a size suitable for making cigarettes. Excipients can be added to give the product a characteristic flavour and ensure that it remains stable over time.

A further subject of this invention is therefore the use of the tobacco leaves obtained with the process according to the invention to make cigarettes, and the cigarettes containing said leaves.

The process according to the invention is particularly advantageous because it allows the desired nicotine content to be obtained regardless of the initial content, with no need to blend different types of tobacco or use expensive rib processing techniques.

The final nicotine content can also reach particularly low values, lower than those obtainable by the traditional techniques used to manufacture cigarettes with a low nicotine content.

The process according to the invention also reduces the chloride content, allowing the use of all the leaves of the tobacco plant and of tobaccos which would otherwise be unusable because they are not combustible.

The invention will now be illustrated with some examples.

# **EXAMPLES**

5

10

15

20

25

Example 1 - Obtaining tobacco leaves with a pre-determined nicotine content of  $0.2\% \pm 0.04\%$  starting with 3 different batches of tobacco with variable nicotine contents. 10 Kg of Virginia Bright tobacco leaves, batch A (0.96% nicotine), is placed in a perforated rack in a stainless steel extractor and extracted with 160 kg of 50° proof ethanol, maintained in continuous recirculation for 4 hours. The extraction temperature, maintained constant by the external jacket of the extractor in which hot water circulates, is 60°C. When the extraction cycle has been completed, the solvent is removed and drying is performed by placing the leaves in drying ovens for a time sufficient to bring the product to a moisture content of 13%, usually 36-48 hours.

10 Kg of Virginia Bright tobacco leaves batch B (1.89% nicotine) is placed in a perforated rack in a stainless steel extractor and extracted with 200 kg of 50° proof ethanol, maintained in continuous recirculation for 8 hours. The extraction temperature, maintained constant by an external jacket of the extractor in which hot water circulates, is 60°C. When the extraction cycle has been completed, the solvent is removed and drying is performed by placing the leaves in drying ovens for a time sufficient to bring the product to a moisture content of 13%, usually 36-48 hours.

10 Kg of Virginia Bright tobacco leaves, batch C (2.24% nicotine), is placed in a perforated rack in a stainless steel extractor and extracted with 200 kg of 50° proof ethanol, maintained in continuous recirculation for 8 hours. The extraction temperature, maintained constant by the external jacket of the extractor in which hot water circulates, is 60°C. After the first 8 hours

5

10

the solvent is eliminated, and a second extraction is performed with 200 kg of water, maintained in continuous recirculation for a further 8 hours. The extraction temperature, maintained constant by the external jacket of the extractor in which hot water circulates, is 40°C. When the extraction cycle has been completed, the solvent is removed and drying is performed by placing the leaves in drying ovens for a time sufficient to bring the product to a moisture content of 13%, usually 36-48 hours.

PROCEDURE	% of nicotine before treatment	% of nicotine after treatment
<b>A</b>	0.96%	0.23%
B	1.89%	0.21%
C	2.24%	0.21%

Example 2 - Analytical data on nicotine content

Sample	Nicotine
Tobacco not subjected to the standardization process	1.271%
Tobacco not subjected to the standardization process and formulated with the excipients	1.183%
Tobacco subjected to process standardization process	0.161%
Tobacco subjected to the standardization process and formulated with the excipients	0.148%
Cigarette with tobacco not subjected to the standardization process	0.92 mg
Cigarette with tobacco not subjected to standardization process and formulated with the excipients	0.81 mg
Cigarette with tobacco subjected to the standardization process	0.135 mg
Cigarette with tobacco subjected to the standardization process and formulated with the excipients	0.125 mg

The nicotine in the tobacco was determined according to the ISO 2881 method.

The nicotine in the cigarette was determined according to the ISO 4387

# method.

Example 3 - Sample formulas of cigarettes made by the process according to the invention

Ingredients	%
Tobacco leaves subjected to the standardization process	85.3
Propylene glycol	3.5
Liquorice	3
Honey	3
Mint flavouring	3
Sugar cane	2
Guar gum .	0.2

5

Ingredients	%
Tobacco leaves subjected to the standardization process	89
Honey	4.4
Glycerol	2.5
Vanilla flavouring	2
Liquorice	0.8
Cocoa	8.0
Tragacanth	0.5

Example 4 - Reduction of chloride content and combustibility

· Cigarette	Tobacco	Result of analytical smoke	% chloride content
A	Virginia Bright Batch 2B012	Negative	1.2
В	Virginia Bright Batch 2B012 Subjected to the process described below	Positive	0.6

## Process:

100 Kg of Virginia Bright tobacco leaves, batch 2B012, is placed in a perforated rack in a stainless steel extractor and extracted with 1000 kg of water, maintained in continuous recirculation for 3 hours. The extraction temperature, maintained constant by the external jacket of the extractor in which hot water circulates, is 40°C. When the extraction cycle has been completed, the water is removed and drying is performed by gentle heating (35°C) and application of vacuum for a sufficient time to bring the product to a moisture content of 13%, usually 36-48 hours.